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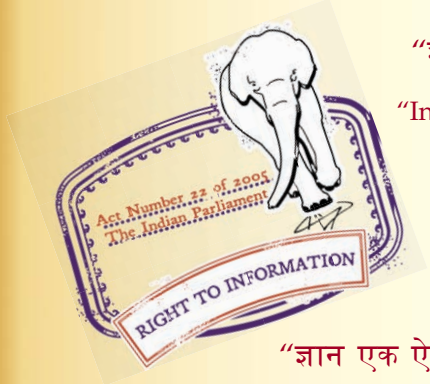
IS 6259 (1971): Anthraquinone, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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SPECIFICATION FOR ANTHRAQUINONE, TECHNICAL

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

January 1972

AMENDMENT NO. 1 JUNE 1973

TO

IS : 6259-1971 SPECIFICATION FOR ANTHRAQUINONE, TECHNICAL

Corrigendum

(*Page* 7, *clause* **A-2.2.1**, *line* 3) — Substitute '100 percent' *for* '10 percent'.

(CDC 46)

Indian Standard

SPECIFICATION FOR ANTHRAQUINONE, TECHNICAL

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(*Continued on page* 2)

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(*Continued from page* 1)

Panel for Anthraquinones, CDC 46 : P 7

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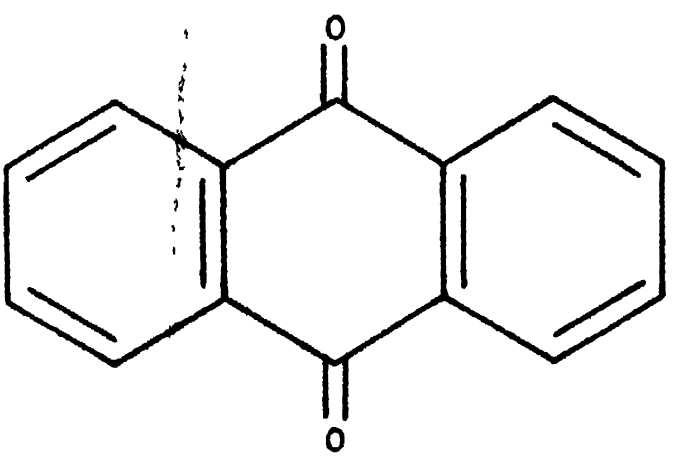
Indian Standard

SPECIFICATION FOR ANTHRAQUINONE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 23 August 1971, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 Anthraquinone ($C_{14}H_8O_2$) is a very important dye intermediate used extensively in the manufacture of vat dyes, disperse dyes and wool dyes. It finds application in textile printing industry, some pharmaceutical preparations and also as pesticide. It is represented by the following structural formula:



ANTHRAQUINONE
(Molecular weight = 208)

0.3 Anthraquinone is a flammable substance and constitutes a slight fire hazard. The flash point is 185°C. Anthraquinone dust is readily flammable and suitable precautions should be taken. Prolonged exposure to anthraquinone dust may cause allergic symptoms.

0.4 This standard is one of a series of Indian Standards on dye intermediates. A complete list of such standards is given on page 8.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with

IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for anthraquinone, technical.

2. REQUIREMENTS

2.1 **Description** — The material shall be in the form of a pale yellow to beige coloured light crystalline powder and shall be free from visible impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR ANTHRAQUINONE, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in)	
			Appendix A	IS : 5299-1969*
(1)	(2)	(3)	(4)	(5)
i)	Moisture content, percent by weight, *Max*	0·2	—	9.1 or 9.3
ii)	Sulphated ash, percent by weight, *Max*	0·5	—	11.2
iii)	Melting point, °C	Shall melt within the range of 3°C including 282°C	—	8
iv)	Anthraquinone content, percent by weight, *Min*	98·0	A-2	—

*Methods of sampling and tests for dye intermediates.

3. PACKING AND MARKING

3.1 **Packing** — The material shall be suitably packed in polythene bags or paper bags or as agreed to between the purchaser and the supplier.

3.2 **Marking** — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer;
c) Lot or batch number;
d) Tare, net and gross weights; and
e) Recognized trade-mark; if any.

*Rules for rounding off numerical values (*revised*).

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Preparation of Test Samples — The material shall be sampled according to the procedure given in 3 of IS : 5299-1969*.

4.2 Number of Tests — Tests for the determination of all the characteristics specified in this standard shall be conducted on the composite sample.

4.3 Criteria for Conformity — For declaring the conformity of the lot to this standard, test results for each of the characteristics shall satisfy the relevant requirement specified.

APPENDIX A

[*Table* 1, *Item* (iv)]

METHODS OF TEST FOR ANTHRAQUINONE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF ANTHRAQUINONE CONTENT

A-2.0 General — The following two methods have been prescribed for the determination of anthraquinone content. The first method (Method A) is the referee method which shall be used in case of any dispute, whereas the second method (Method B) is for the routine analysis.

*Methods of sampling and tests for dye intermediates.
†Specification for water, distilled quality (*revised*).

A-2.1 Method A

A-2.1.1 *Principle* — In this method the anthraquinone is reduced to its leuco derivative by heating to 60 to 80°C with an aqueous solution of caustic soda and sodium hydrosulphite. The sodium salt of leuco anthraquinone forms a bright red coloured solution and is filtered through a sintered glass funnel. The leuco anthraquinone is oxidized back to anthraquinone by the addition of hydrogen peroxide or by aeration. The precipitated anthraquinone is filtered through a sintered glass crucible washed free of alkali, dried and weighed.

A-2.1.2 *Procedure* — Prepare a solution of 10 g of sodium hydrosulphite (*see* IS : 1919-1961*) in 100 ml of 10 percent sodium hydroxide. Allow to stand for one hour and filter through a filter paper.

A-2.1.2.1 Weigh accurately about 1 g of the powdered anthraquinone and transfer it into a 250-ml conical flask. Add 100 ml of water and 50 ml of the hydrosulphite solution prepared as above. Heat the mixture for 10 minutes in a water-bath at 60 to 80°C. There results a bright red coloured solution of leuco anthraquinone. Filter through a G2 sintered glass crucible; taking care not to allow oxidation of the material on the crucible or foaming of the filtrate (do not apply too high a suction for filtration). Before starting filtration, put a little diluted (10 times) hydrosulphite solution on the filter. See that the crucible is never empty. Treat the undissolved anthraquinone remaining in the flask with dilute (10 times) hydrosulphite solution at 60 to 80°C. Reduction is complete when the filtrate is free of any tinge of red.

A-2.1.2.2 To the filtrate at 60 to 80°C, add 10 ml of 30 percent hydrogen peroxide to oxidize back the anthraquinone. Oxidation is complete when there is no red colour. Oxidation may also be carried out by passing a slow current of air through the leuco solution at 60 to 80°C (considerable foaming occurs in aeration). The precipitate is filtered through a tared G4 sintered glass crucible, washed free of alkali with hot water and dried at 100 to 110°C to constant weight.

A-2.1.3 *Calculation*

$$\text{Assay, percent by weight} = \frac{W_2}{W_1} \times 100$$

where

W_2 = weight in grams of the regenerated anthraquinone, and

W_1 = weight in grams of sample taken for the test.

*Specification for sodium hydrosulphite, technical.

A-2.2 Method B

A-2.2.1 *Principle* — This method makes use of the great stability of anthraquinone to the action of sulphuric acid. Anthraquinone is dissolved in 10 percent sulphuric acid and held at 95 to 100°C for 10 to 15 minutes. The impurities in anthraquinone are digested by this process. Anthraquinone is reprecipitated by allowing the solution to absorb water gradually over 12 hours. It is then filtered, washed free of acid and dried.

A-2.2.2 *Procedure* — Weigh accurately about 1 g of anthraquinone and place it in a porcelain dish of 20 cm diameter. Carefully pour 10 ml of 100 percent sulphuric acid in order to cover all anthraquinone crystals. Put the dish on a water-bath at 95 to 100°C. Maintain for 10 to 15 minutes. Remove from water-bath, keep in a bell jar containing water. Allow to absorb moisture over 12 hours. Drown the contents of the dish into 200 ml water contained in a 400-ml beaker, washing down the dish well. Filter through a tared G4 sintered glass crucible and wash with distilled water till the filtrate is neutral. Then, wash with 200 ml of boiling potassium hydroxide solution (0·1 g dissolved in 100 ml) and finally with boiling distilled water till free of alkalinity. Dry at 100 to 110°C to constant weight.

A-2.2.3 *Calculation*

$$\text{Assay, percent by weight} = \frac{W_2}{W_1} \times 100$$

where

W_2 = weight in grams of reprecipitated anthraquinone, and

W_1 = weight in grams of sample taken for test.

INDIAN STANDARDS

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4265-1967	4-4′ Diaminostilbene 2-2′ disulphonic acid	4·00
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